From these results it can be concluded that mixtures of acetone and n-butane can be analyzed quantitatively by low voltage mass spectrometry.

Minutes of

SUPERVISORS' MEETING

April 10 - 11, 1958

Present: Mr. A. E. O'Keeffe (Chairman Administrative)
Mr. L. L. Long (Secretary)
Dr. L. S. Harrow (Chairman Technical)
Dr. R. C. Sproull
Mr. H. J. Dooley
Mr. J. Y. Mason, Jr.
Mr. F. E. Resnik
Mr. W. H. Danker
Mr. F. L. Gager, Jr.
Mr. J. T. Butler
Mr. S. W. Pleasants
Mr. C. C. Cosby, Jr.
Mr. A. P. Super
Mr. J. E. Hardcastle
Dr. C. V. Mace
Mr. J. C. Holmes
Mr. G. H. Burnett

ADMINISTRATIVE

Mr. O'Keeffe requested that each group make every effort to anticipate his supply needs so that our purchasing can be done in a more orderly manner.

Mr. Cosby reported that the new laboratory area would be ready for initial occupancy by April 13th or 14th.

The project cost system was discussed. Mr. Dooley emphasized that the time cards should be sent to Mr. Cosby by noon each Monday. It was decided that each supervisor should check and initial the cards for those in his group prior to sending them to Mr. Cosby.

The handling of project notebooks under the new project system was discussed. A discision was made that several projects could be recorded in one notebook provided that all project data appearing in a notebook be properly indexed inside the cover prior to turning the notebook in for filing.

Mr. Dooley reported that a new, improved interview form had been developed and is ready for use.

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Source: https://www.industrydocuments.ucsf.edu/docs/hkxk0000

SiO_2	12%
$CaCO_3$	38
Isobutylene rubber (Vistanex)	23
Isobutylene rubber + natural rubber	15 (less than 5% natural rubber)
Plasticizer (similar to type RBH-510; a natural resin)	12

TECHNICAL

21-0101 Chemical Composition of the Gas Phase of Smoke (Murrill)

The gas phase of smoke was chromatographed on silica at -70°C, and a small peak corresponding to the retention time of NO_2 was trapped. Infrared analysis was not possible because of insufficient sample. When known NO was chromatographed and the peak trapped, an excellent IR spectrum was obtained. However, a known gas, indicative of NO_2, was observed at room temperature and a blue color at -195°C. The latter is indicative of N_2O_3 ($NO + NO_2$). It is thought that small amounts of air are oxidizing a portion of the NO to NO_2. Additional work will be carried out to develop techniques of handling NO without conversion to NO_2. The possibility of NO existing in smoke is considered remote because of its chemical activities.

21-0102 Chemical Composition of the Particulate Phase of Smoke (Poindexter)

A basic fraction from the particulate phase of smoke has been chromatographed on silica gel. Fluorescent and yellow colored materials have been eluted free of alkaloids. Fractions are being analyzed instrumentally and will be subjected to additional separational methods in attempts to identify the components.

Discussion: Dr. Harrow suggested that a method for phenols in smoke could utilize part of the distillate obtained during the nicotine determination. The nicotine is steam distilled over from an acid solution.

21-0201 Qualitative and Quantitative Analyses of Tobacco Leaf Compounds (Carpenter, Edmonds, Greene, Brunot)

A. Petroleum Ether Extract of Tobacco: A procedure for determining the saponification number is being adapted for small samples. This will be used for chromatographic fractions obtained from adsorption chromatography of petroleum ether extracts.

WIN extract, equivalent to 25 pounds of filler, has been subjected to steam distillation. This extract is being used as a model before using petroleum ether extract of leaf. The leaf extract will be separated with acid, basic, and neutral fraction before steam distillation. The steam volatile compounds should include those materials which contribute to aroma. The WIN extract should give fractions which will be large enough to allow additional fractionation. Techniques developed working with it should be applicable to the petroleum ether extract.

B. Polyphenols: A sample of aged tobacco previously extracted with petroleum ether has been extracted with ethanol. The extracts have been compared and one phenolic zone is present in the petroleum ether solution. Its chromatographic mobility

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Source: https://www.industrydocuments.ucsf.edu/docs/hkxk0000

studies are being made. Moisture loss from the salt at elevated temperatures is being studied.

Filler improvement by the addition of a mouth emollient has been tentatively established in principle by a panel test. PM's containing 7% propylene glycol were reported by 11 of 18 judges to be sweeter and significantly less irritating. This cigarette is too moist for present production. Cigarettes with lower percentages of propylene glycol have been made and will be tested.

is the same as one of the components in the 95% ethanol. The fluorescence under ultraviolet light differs slightly from the component found in ethanol. A band believed to be chlorogenic acid has been eluted and analyzed by ultraviolet spectroscopy. The absorption in an acid and neutral agrees with known chlorogenic acid. The peak in base is displaced by about 20 millimicrons. The known will be subjected to the same conditions as the sample from paper since chlorogenic acid is unstable especially as the sodium salt. The length of time the sample stood in base may be critical.

C. Alkaloids: A group of eight Turkish tobacco samples have been obtained from Mr. Mason. These will be analyzed by the chromatographic-spectrophotometric nicotine method. A "spectrum of qualities" and nicotine contents are covered by the samples.

D. Carbohydrates: A statistically designed experiment is being carried out with samples of fructose, glucose and rhamnose alone and in mixtures. The cysteine and cysteine-carbazole colorimetric determinations are being evaluated. Initial results indicate considerable variation in the glucose determinations.

A "green" Turkish sample of tobacco has been submitted by Mr. Mason. Green leaves were hand-picked from a large sample of the tobacco. An aqueous extract of the tobacco will be examined qualitatively for the sugar components.

Discussion: In connection with this work, Mr. Butler reported when known amounts of glucose are added to tobacco, the recovery is not complete.

21-0104 Chemical Composition of Whole Smoke - Formaldehyde (Carpenter, Martin)

The colorimetric method for formaldehyde in smoke gives reproducible values for replicate samples from the same smoke collection. The effects on the method of variations in the trapping, the smoking, and the cigarettes themselves are being determined. Cigarettes (PM regulars), selected on the basis of weight range, puffability, and firmness, are being used to eliminate as much as possible the variation between cigarettes.

22-0105 Physical Mechanism of Smoke Formation (Holmes)

Major effort has been toward measurement of the temperature of exit smoke from Kent and Hi-Fi Parliament. These studies have shown that the overall average temperature of Kent smoke is lower than that from Hi-Fi Parliament by about 5°F. Data is insufficient to show difference of temperature with butt length. It should be noted that the temperature of exit smoke does not go above room temperature until the butt length is shorter than 30 mm. When a butt length of 20 mm is reached temperatures are in the range of 140-150°F.

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Source: https://www.industrydocuments.ucsf.edu/docs/hkxk0000

Varieties from the 1957 N. C. State Official Variety Test.

The technique of measurement of temperature profiles has been improved so that temperature isotherms can be located without using the char line as a reference point. The new technique uses three thermocouples and should eliminate some of the variation now encountered because of the uncertainty of locating the char line. Only a few determinations have been run by this method to date. The results are encouraging, however, there is still a wide variation in the maximum temperatures encountered.

Discussion: It was suggested that the heat capacity of the filters affects the exit smoke temperature. Mr. Holmes stated that the heat capacity taken into consideration when investigating new filter media.

Dr. Sproull asked Mr. Holmes to work with Mr. Super on possible new materials designed for both filtration and exit smoke temperature effects.

23-0101 Instrumentation. Chemical Composition of the Gas Phase of Smoke

I. Development of Methods for Determining Quantitatively the Gas Phase Constituents of Smoke (Varsel, Bell, Dodson)

Work has continued on the development of a method to reproducibly collect and analyze the gas phase of cigarette smoke. The nitrogen-flushing smoking apparatus is being perfected in regard to operational procedure; particular emphasis is being placed on trapping efficiency. Plans are to study the effect of $Ba(OH)_2$ on the various smoke constituents.

II. Instrumentation Service Qualitative Identification of Materials (Bill, Vilcins)

The infrared spectrophotometer is being used to monitor the gas chromatographic separation of the oxides of nitrogen because of the infrared's specificity for these materials. These materials are so reactive that they have been attaching the NaCl faces on the gas cell and forming $NaNO_3$.

23-0201 Bases (Resnik)

The usual quantitative nicotine analyses were performed with the ultraviolet spectrophotometer for Mr. Edmonds.

A fraction of the ethanol extract of tobacco separated by Mr. Edmonds was analyzed with the ultraviolet spectrophotometer. The acid and neutral curves were identical for chlorogenic acid but the basic curve was different than the curve of the reference chlorogenic acid. This will be investigated further.

23-6101 Service Electronics (Morrell, Resnik)

The usual maintenance and service work was performed by the Electronics Group during this period.

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Source: https://www.industrydocuments.ucsf.edu/docs/hkxk0000

further work.

The investigation into synthetic filler from carbon has been started.

23-6001 Instrumentation. Instrument Design and Development and Adaptation. Modifications of the Mass Spectrometer

I. Stabilization of the Magnetic Field (Morrell, G. Smith)

Work is continuing on the stabilization of the mass spectrometer electromagnetic field. Two matched bismuth grids have been received and it is planned to mount these grids in the cell. The effect of temperature differences between these grids will be investigated.

II. Isatron temperature Control (Morrell, G. Smith)

The substitute reference voltage supply was found to be inadequate and the original reference heater was replaced in the isatron temperature control chassis. A slight modification was made to provide greater ease of adjustment for the reference heater control.

Plans are to use a direct isatron temperature control device such as a calibrated controlling pyrometer. Efforts will be made to obtain a precise, adjustable temperature controller with an accuracy of ±0.25°C.

23-6005 Instrumentation. Instrument Design and Development and Adaptation. Modifications of Chromatographic Devices.

I. Ionization Gauge (Morrell, G. Smith)

Future work with the ionization gauge is planned with the ultimate objective the construction of a unit for high temperature operation as a gas chromatography detector.

Since electrode temperatures are more indicative of gauge operation than ambient or inner space temperatures, a special design is planned for the study of high temperature effects. Thermocouples will serve the purpose of indicating temperatures and of conducting gauge current. With the thermocouple welded directly to the electrode, true temperatures of the cathode and anode will be obtained.

Discussion: In connection with the operation of the ionization gauge at temperatures above the inlet gas temperature, Mr. Holmes pointed out that you will get a pressure differential which will affect the ionization thus giving erroneous readings.

23-6201 Instrumentation. Research with Instruments. Mass Spectrometer (Varsel, Bell, Dodson)

Experiments were carried out to determine if two compounds, which have the same molecular ion but different appearance potentials, can be analyzed quantitatively by low voltage mass spectrometry.

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Source: https://www.industrydocuments.ucsf.edu/docs/hkxk0000

Storage testing of cigarettes in air, oxygen, nitrogen, and vacuum has been started. It is now thought that the evacuation period before introduction of the gases into the desiccator may have impared the flavor of the cigarettes at the start of the test. With this in mind, a fresh start will be made. The desiccator will be flushed, then after a short evacuation period, the gas to be used in the storage test will be introduced.

Four mixtures of n-butane and acetone were analyzed at 9.6 volts. Acetone ionizes at this voltage while n-butane requies a higher voltage for ionization. The results of these analyses are as follows:

% Acetone Added	% Acetone Found
50	54
40	53
50	53
50	53

Further fundamental studies of this type will be undertaken.

23-6203 Instrumentation. Research with Instruments. Ultraviolet Spectrophotometer (Kuhn)

No New experimentation has been carried out on this project during this period.

23-6202 Instrumentation. Research with Instruments. Infrared Spectrophotometer (Vilcins, Bill)

Work was continued to minimize the high blank obtained by eluting filter paper with water in order that the micro KBr technique could be applied to the identification of compounds eluted from paper chromatograms.

Four pieces of Whatman #1 filter paper were washed with 2% acetic acid for 24 hours and the papers dried under vacuum. Infrared analysis of the capillary action, aqueous washings of these papers showed a minimum, reproducible background absorbance.

Plans are to wash several large pieces of paper with 2% acetic acid and use these papers for the separation of carbohydrates from tobacco by paper chromatography. Attempts will be made to elute the isolated carbohydrates and identify these materials by infrared spectroscopy. This work will be carried out jointly with the Organic Section.

Work has been initiated on improving the infrared analysis of small amounts of liquids. Methods of increasing the sensitivity of "film analysis" are being investigated.

23-9201 Instrumental Services (Bill, Bell, Dodson, Kuhn, Morrell, C. Smith, Varsel, Vilcins)

Service to Polymer Industries: A sample of a competitor's latex adhesive was submitted for infrared examination by Polymer Industries. Thus far, the filler has been identified as $CaCO_3$ and SiO_2 and the rubber as an isobutylene polymer (Vistonex). The plasticizer has not been isolated as yet.

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Source: https://www.industrydocuments.ucsf.edu/docs/hkxk0000

The design for the Vari-speed drive frame is complete and a drawing is being prepared.

Bids have been received for the rotating seal assembly on the ordering cylinder. The bids are excessively high, so the seal will be redesigned.

24-8104 Flavor (Danker, Maxwell)

New York Project 1018 New Flavored Cigarette: The best combinations of our top four flavors to date and examples of some new promising flavors were shown to Mr. Latham, Mr. Early, and the representatives of the advertising agency who are to handle this project. The following selling points were suggested and examples shown:

1. Flavor which comes through into the mouth (shown by 10% smoke dilution).

2. Mouth Watering Effect (shown by smoking).

3. Increased mildness by increased moisture (by adding water as is or as a dry generator).

4. Mouth emollient effect (of liquids such as TEG and propylene glycol). This had been suggested by inert filler and plug impregnated additives in earlier experiments.

5. Flavor on plugs lasts longer (is not burned in filler).

A spearmint-peppermint combination proved most acceptable by panel tests with an anethole-fruit combination next best. Both were shown to Mr. Hatcher and Mr. Britton and later discussed and demonstrated to our New York group and the full agency group. The flavors were added to Parliament plugs with and without menthol and to Spud plugs. The latest conclusions are to use a low menthol-low flavor type. We determined the amount of flavor added to the plug for the mint plus menthol on Hi-Fi. The first factory batch proved to be close to our lab made winner. Mr. Hatcher expressed satisfaction with progress made.

Hydrated aluminum sulfate is showing promise in making milder smoke when added to filler. Some judges found a metallic off flavor. This is being checked by taste and chemical means. Instrument tests are also scheduled.

Emollient mount effect will be further tested. Does it really reduce tongue, throat, nose irritation? Appreciably?

Filler and plug impregnated flavor are being compared first puff vs. fourth puff vs. eighth puff to determine which gives more flavor at what puff.

An additional series of flavors is being screened for solubility, stability, mouth effect and appearance. It is hoped that we may select about eight more flavor combinations in this way.

About ten flavors -- all different -- all with a good acceptability shall then be submitted to interlocked comparisons in designed experiments. These will be reduced to a preferred

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Source: https://www.industrydocuments.ucsf.edu/docs/hkxk0000

other methods were as follows: photographing of a cross-section without the waxing; separation of the fibers by hand, then visual counting.

Mr. O'Keeffe pointed out that the U. S. Testing Laboratories have a standard mold technique for fiber count and dimensions that is very good. This should be looked into.

few for tests to be made by Jet Lincoln. A maximum of internal testing is planned for security reasons.

PM Booster Program: Mr. Latham has asked us for suggestions to boost PM sales. We have started a sensory evaluation program to determine differences between PM and its non-filter competitors. All differences -- odor, appearance, feel -- are being determined by panel analysis then obvious differences will be further established statistically and reported to Mr. Latham. The technique previously developed for sensory brand analysis will be utilized for this effort.

Philip Morris Cigarette Shelf Life Flavor Changes: Our first series of tests show that a detectable pack aroma reduction occurs in 4 weeks. This was found to be greatest for Hi-Fi Parliament less for Marlboro.

Study of Fillers: This program was continued. 20% BL was found difficult to detect in PM blends. Mr. Hatcher was told of our findings on this and on the tests of Herty stem blends in which 30% stems was a near threshold level. Mr. Hatcher is developing a Hi-Fi variation of Spud filler for the 1018 project.

Discussion: As part of the study of the effects of aluminum sulfate on the smoke flavor, Mr. Long suggested a complete investigation into the effect of added aluminum sulfate on filling power (i.e. cigarette weight reduction) and smoke solids.

25-0203 Correlation of Chemical Composition, Physical Properties, and Smoke Analysis with Leaf Quality (Bass, Sharp, Mason)

Chemical and smoke analyses are in progress on flue-cured and Turkish tobacco samples.

Results from the samples analyzed indicate that the evaluation based on the leaf analysis is an indication of the nicotine and smoke solids content of the smoke. Those tobaccos having large contents of nicotine, total nitrogen, T. V.B. and petroleum ether extract and small sugar contents have large nicotine and smoke solids contents in the smoke.

Correlation coefficients are being calculated.

25-0204 Development of Improved Leaf (Bass, Sharp, Mason)

The chemical composition of the test tobaccos included in the 1957 Official Variety Test have been compared to the Hicks control. McNair 121, Bell 15, Coker 1, and Coker 3 were the middle type tobaccos; P.D. 83 and P.D. 97 heavy and strong; and Virginia 617, Virginia 680, and Vesta 5 similar to Hicks.

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Source: https://www.industrydocuments.ucsf.edu/docs/hkxk0000

...for use with the Burley machine.

35-4003-00 Low Tar Cigarettes (Hind, Burnett)

Extraction of PM filler by one treatment with cold acetone removed about 4.5% by weight of material. Cigarettes prepared from this material on the "Chico" maker were mild and gave a pleasant smoke.

McNair 121 and Bell 15 were released last fall. The appearance of these tobaccos is similar to Coker 139. Neither appears to be as desirable as Hicks. The Bell 15 is more desirable than McNair 121.

The results of the evaluation of the Coker Seed Company tobacco have been submitted to the Leaf Department, report April 4. The Coker 187X Hicks was very desirable, Hicks ESR not desirable because of its excessive strength, and the remaining lines were excessively mild.

25-0702 Procedures and Conditions for Accelerated Aging (Crayton, Mason)

Georgia tobacco, GHC, with a moisture content of 12.5% was aged at 115°F. The color of this tobacco after aging was good. There was a significant decrease in nicotine, T.V.B., sugar, and ash contents. The aroma of this tobacco was not as good as that aged at 14% moisture content.

Cigarettes have been prepared and submitted for smoke analysis and taste panel tests.

The dehumidifier on the Hotpack cabinet continues to give trouble. A service man from Hotpack will come down to correct this.

25-8401 Services to Leaf Department (Bass, Mason)

Several samples of flue-cured and burley dealers' strips from W. L. Robinson and Dibrell Bros. have been evaluated. The results have been submitted to the Leaf Department.

The analysis of the 1957 stemmery samples of flue-cured tobacco is complete. Grade averages by belts are being completed.

The overall average for all grades and belts shows that the 1957 tobacco had larger contents of nicotine and T.V.B. than the 1956 crop.

32-0306 Filling Power Studies (Burnett)

Insolubles and filling power results were reproducible, by grade, from hogshead to hogshead with reasonable accuracy, irregardless of the year's crop or stemmery.

33-1304 ATBL Studies (Westermann, Clarke)

Work has continued on further improving the smoking quality of ATBL. ATBL consisting of 40% stems (with no cooking) were smoked at 100% level by the flavor department and were found to be mild and non-irritating with no apparent BL taste. The most pronounced taste was that of vanillin.

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Source: https://www.industrydocuments.ucsf.edu/docs/hkxk0000

71-1306 Physical Testing of BL Products (Wickham, Stewart, Pleasants)

No production BL was received for testing during this period.

One sample of ATBL evaluated for its physical characteristics was slightly below the desired tensile strength and slightly high in breakage. The material was very low in dust loss. These

Methods for processing stems, other than cooking, have been investigated. (The cooking of stems appears to give a slight obnoxious taste to the ATBL.) The non-cooking methods investigated include treatment of stems with nitric acid, cold digestion of stems in ammonium carbonate, and mechanical treatment of rolled stems which had been cooked in water for 16 hours. The physical properties (tensile strength, dust loss, and breakage) of ATBL containing uncooked stem pulp is not as good as the material containing cooked pulp.

33-5001 Packaging (Carr, Super)

Continuing moisture protection tests of cup packages with coated foil and K-type cellophane, packs were stored at 75°F, 65% R.H. for four weeks, then at 90% R.H., 90°F for five week. The results showed that the special cellophane increased the moisture protection of the pack and the special foil had no effect. Previous results at other conditions had shown no differences among the packs tested.

Investigations are underway to determine if Cryovac (Poly vinylidene) bags would be superior to polyethylene bags for shipping tobacco filler.

Tests in which cigarettes are to be stored under vacuum, air, oxygen, and nitrogen can not be started for two weeks because smoke tars and nicotine can not be determined until then.

34-0501 Microscopic Studies (Johnson, Clarke)

By staining techniques we were able to identify the materials in Scotch-Pak (a wrapping material manufactured by Minnesota Mining) for Milprint. The material contained two layers of polyethylene, a thin layer of metal, and a layer of mylar. Samples of known materials were used as controls.

The old and new L & M and Viceroy cigarette filters were examined microscopically for the Physical Testing Group to determine material content and the number of filaments in the filters.

Eight different types of cigarette papers were examined and compared with Kent's paper to see which type more nearly resembled it and if there was any way to determine slow and fast burning papers microscopically. This was done for the Methods Development Section.

34-1301 Improvement in BL as Manufactured (Scherr)

Particle Size Studies: Physical evaluation of three runs using particle size material of -30 mesh, -50 mesh, and -80 mesh showed that as the particle size increased the sheet weight increased. Dust loss and breakage also increased and the color of the material produced was lighter in color than standard production BL sheet.

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Source: https://www.industrydocuments.ucsf.edu/docs/hkxk0000

for nine brands of the Comparison of Brands pickup for March.

Griffith Steam Distillation: During this period 402 samples were analyzed with this equipment.

Evaluation of data showed that the sheet weight increased about 25% while dust loss increased from about 4% to between 7% and 10%.

34-3101 Filters (Super)

Filters have been made from five types of papers representing different amounts of pulp hydration so that the effect on filtration efficiency and moisture removal from smoke can be determined. Also filters have been made from Solka-flor (alpha cellulose) and two types of cellulose heated to give it anion exchange properties.

35-0114 Design of Smoking Machines (Wright)

The design of a totally enclosed smoking cabinet for use with the 20 port smoking machines has been completed. Drawings will be submitted to fabricators for bid this week. The design of installation apparatus for the cabinet is underway.

35-1304 Development of an All Tobacco BL (Michels)

Mr. Edwardes suggested a higher pulp freeness of material would be desirable in the formation of an all tobacco BL material. In connection with the above suggestion a batch of material was prepared by water cooking, then shredding and brushing in a Sprout-Waldron refiner and compared with a batch of material cooked in a 5% ammonium carbonate solution and otherwise prepared the same. In this comparison, microscopic examination showed that the water cooked stems yielded longer fibers than those of the ammonium carbonate cooked stems.

35-2101 Development of New Filter Materials (Michels)

An investigation of methods of etching fibers of different materials showed that the surface area of nylon fibers can be increased by etching with a 50% solution of zinc chloride.

Future work in the investigation of etching processes will depend upon the relative filtration efficiency of the etched fiber compared to the non-etched fiber.

35-2301 Preparation and Extraction of Organoleptic Materials (Hind, Burnett)

We have esterified 890 grams of bright stem solids in ethanol, using sulfuric acid as a catalyst. Part of the product has been worked up to yield 48 grams of a flavoring material having a strong rum-fruit odor. It is probable that besides esterification of some of the available organic acids, other alcoholysis products are being formed. The yield is roughly equivalent to 7% on the weight of bright stems.

We are also trying to obtain a useful material by reducing bright stem solids using sodium amalgam. In a first trial,

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with a standard deviation [illegible], neither the time of smoking, nor the original weight, whether heavier or lighter or average, nor the variance of the individual results of a run, either for zero drying time or after oven drying, could be correlated with the percent change on oven drying.

a strong odor of phenyl acetic acid was noted, and subsequent extraction with ether yielded a residue with a more pleasant flavor. Untreated stem extract yielded a small amount of material with a pleasant odor after prolonged ether extraction.

35-4003 Low Tar Cigarette (Extraction of Filler with Various Solvents) (Hind)

The rate of extraction of materials from PM filler by hexane and methylene chloride at 20°C has been measured. Data has been obtained on batch extractions using hexane, methylene chloride, acetone and chloroform, and work is being done to obtain extraction ratio from acetone and chloroform.

35-4006 Development of Semi-Works for Cigarettes (Wright)

Lay-out and detail drawings for a quick charge gear box for the Chico cigarette making machine are about 80% complete.

Detail drawings of a rotating seal assembly for the spray drum of the flavor drum roller assembly are complete and have been submitted to two jot shops for bid. Bids on the job should be received this week.

Other work done on the Chico machine include the redesign of paper rolls one of which has been installed and drawings for a bracelet and two other paper rollers have been completed.

Routine experimental cigarettes have been made up per request of various R & D personnel.

71-0204 and 71-0304 Leaf and Filler Analyses (Nedlock, Wiley, Pleasants)

The following analyses were completed on leaf and filler during this period:

134 Nicotine (Perchloric): Determinations were made on routine stemmery samples, product audit cigarettes from the Louisville and Richmond factories, special test cigarettes from the Overseas Department and the Marketing Department and Comparison of Brands cigarettes for February 19th pickup.

126 Total Ash and 126 Petroleum Ether: The samples analyzed were Comparison of Brands cigarettes, routine stemmery samples, and priority leaf samples.

59 T.V.B.: Determinations were made on routine stemmery samples, Turkish priority samples, Comparison of Brands cigarettes and aging samples received from Mr. Crayton.

306 Total Nitrogen: Determinations were made on stemmery samples, priority Turkish and product audit samples, aging samples and Comparison of Brands cigarettes.

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Discussion: Dr. ...rrow stated that the moisture in the particulate phase of smoke could be determined with the Dean Stark apparatus.

It was pointed out that Battelle determines tars with the ultraviolet spectrophotometer.

219 Sugar (Reducing): The samples analyzed were product audit cigarettes, special test cigarettes, routine stemmery samples, priority samples and Comparison of Brands cigarettes.

826 Moistures: Determinations were made on special test cigarettes, Comparison of Brands cigarettes and stemmery samples.

Total Samples Analyzed: 1797

71-1306 Physical Testing of BL Products (Wickham, Stewart, Pleasants)

The physical characteristics of three (3) samples of pilot plant BL were evaluated during this period.

One (1) sample of production BL was evaluated for its physical characteristics also.

71-0109 Smoking Machines (Wickham, Stewart, Pleasants)

The following number of smoke analyses was completed during this period: 318 tar and 636 nicotine.

In a further effort to establish that there is a relationship between tar delivery and moisture content of cigarettes, a study was made where to 50 ml aliquots of a composite tar sample, a 0.1 ml of H_2O was added to 9 dishes and 0.7 ml of H_2O was added to 9 dishes. The average of the nine results are as follows:

0.1 ml of H_2O	0.7 ml H_2O
20.40	20.62

From the results obtained, there was a slight difference in the dishes with 0.7 ml. of H_2O added.

A mercury manometer was made and is now being used to check for leaks in the units prior to smoking. This may be one factor which will prevent low ports during smoking therefore reducing some of the variance now present between ports.

71-4007 Comparison of Brands (Wickham, Stewart, Pleasants)

The Comparison of Brands of the February 19, 1958 pick-up has been completed. The report is now being written.

Work is now progressing on the March 19, 1958, pick-up from eight widely dispersed cities.

Old Gold filter (March) has changed to a fast burning paper which has characteristics similar to Ecusta 515 or Schweitzer 135 paper. This change in paper has supposedly reduced the tar delivery of the cigarette. A study is now being conducted to confirm that the tar delivery has been reduced.

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Source: https://www.industrydocuments.ucsf.edu/docs/hkxk0000

71-6902 Methods Improvement (Wiskham, Stewart, Pleasants)

In an attempt to decrease the amount of variation in the smoking analysis, a complete study of the method and technique involved was initiated. This study was devised by our consulting statistician, Mr. Tom Budne, who spent considerable time in discussing what was involved and laying out a plan to test the variables.

Variables introduced into the routine analysis were packaging prior to smoking, extra washing of the filters, sealing the flasks prior to the transfer of the tar solution to tared aluminum pans, extra flask rinses during the transfer from flask to pan, extra steam bath time, desiccating dried tar pans prior to placing them in the oven, and extra oven time.

After six machine loads of twenty (20) ports had been smoked, a study of the results revealed a notable difference in two of the variables introduced into the routine analysis. The steam bath time had an effect. The longer a sample stayed on bath, the lower the amount of tar. Since the steam bath time is determined by the technicians, there is a possibility of variation between tar samples.

The other problem brought out by this experiment was that oven positions gave high and low results depending upon which shelf the pan was placed, top, middle, or bottom, also the left and right sides gave different results as did the back and front positions of the oven.

From two (2) runs of forty (40)ports with only the oven positions controlled, results show the top shelf gives low results and the bottom shelf gives high results with the middle shelf results falling between the results for the top and the bottom. The sample on the left side of the oven had a higher average than the right side as did the average of the front samples which were lower than the average of the samples placed in back.

On one (1) run a draft was created to determine if air currents affected the final results. From this it was found that the average results were lower by about 0.8 mg/ cigarette.

Nicotine (Griffith): A total of 206 samples of smoke solutions were analyzed on this equipment during this period. These samples were analyzed for method evaluation and training purpose.

BL in Cigarettes: The project of picking one pound of Winston reconstituted tobacco has been altered as a result of polling the supervisors to find out exactly what their needs were. The actual quantity of Winston reconstituted tobacco required is about 80 grams. The quantity picked to date is 80 grams, therefore this project will be terminated.

1001502200

Source: https://www.industrydocuments.ucsf.edu/docs/hkxk0000

Mr. Dooley reported that a new, improved [illegible] had been developed and is ready for use.

Petroleum Ether Extraction Time Study: Since the last biweekly report no work has been done on this project. As soon as our priority leaf samples slow down this study will be resumed.

Cellulose in Filter Plugs: A method for the determination of cellulose powder in filter plugs has been developed by the Methods Development Section and turned over to the Analytical Section. To date, five analyses have been made on two cigarette brands for familiarization purposes.

In the analysis, twenty filter plugs (five at a time) are dissolved in acetone. The acetone is drawn off by vacuum leaving behind insoluble cellulose powder. The powder is transferred to a previously tared beaker and weighed to determine the percentage of cellulose powder in the filter plugs.

72-0103 Quantitative Determination of Smoke (Butler)

Both vacuum desiccator and air drying have been tested as a means of conditioning the material collected on the glass filters after smoking. We have only a few results obtained by passing dry air through the filters at a given rate for various times but these have been disappointing. Vacuum desiccator drying has yielded more consistent results with the better results obtained at a point just to the leveling off point of the drying curve. Since a static method has definite advantages from a practical workable standpoint the desiccator drying will be statistically analyzed.

72-0201 Development of Methods (Butler)

I. Development of Method for Acids in Leaf: The R_f values have been determined for five organic acids using a xylene-butanonl(3:2 v/v) solvent system and developed in the bromo phenol blue indicator. The following duplicate R_f values were obtained: citric, 0.14, 0.13; malic, 0.18, 0.19; oxalic, 0.41, 0.42; succinic, 0.53, 0.54; fumaric, 0.77, 0.72. Each of these acids were spotted on a paper strip and the separation was good except for citric and malic.

II. Development of Method for Nitrogen Distribution in Leaf: The experimental work on this method is complete and a completion report will be written for this method after receiving a statistical analysis of the results.

73-0601 New Improved Method of Measurement of Dynamic Burning Time (Butler)

The dynamic burning time for weight-selected cigarettes in the range of 1.02 - 1.06 grams stored at 60% relative humidity is approximately 55 seconds with a two sigma value of approximately ±12 seconds according to the present test method. The cigarettes generally burn unevenly. Future runs will be made

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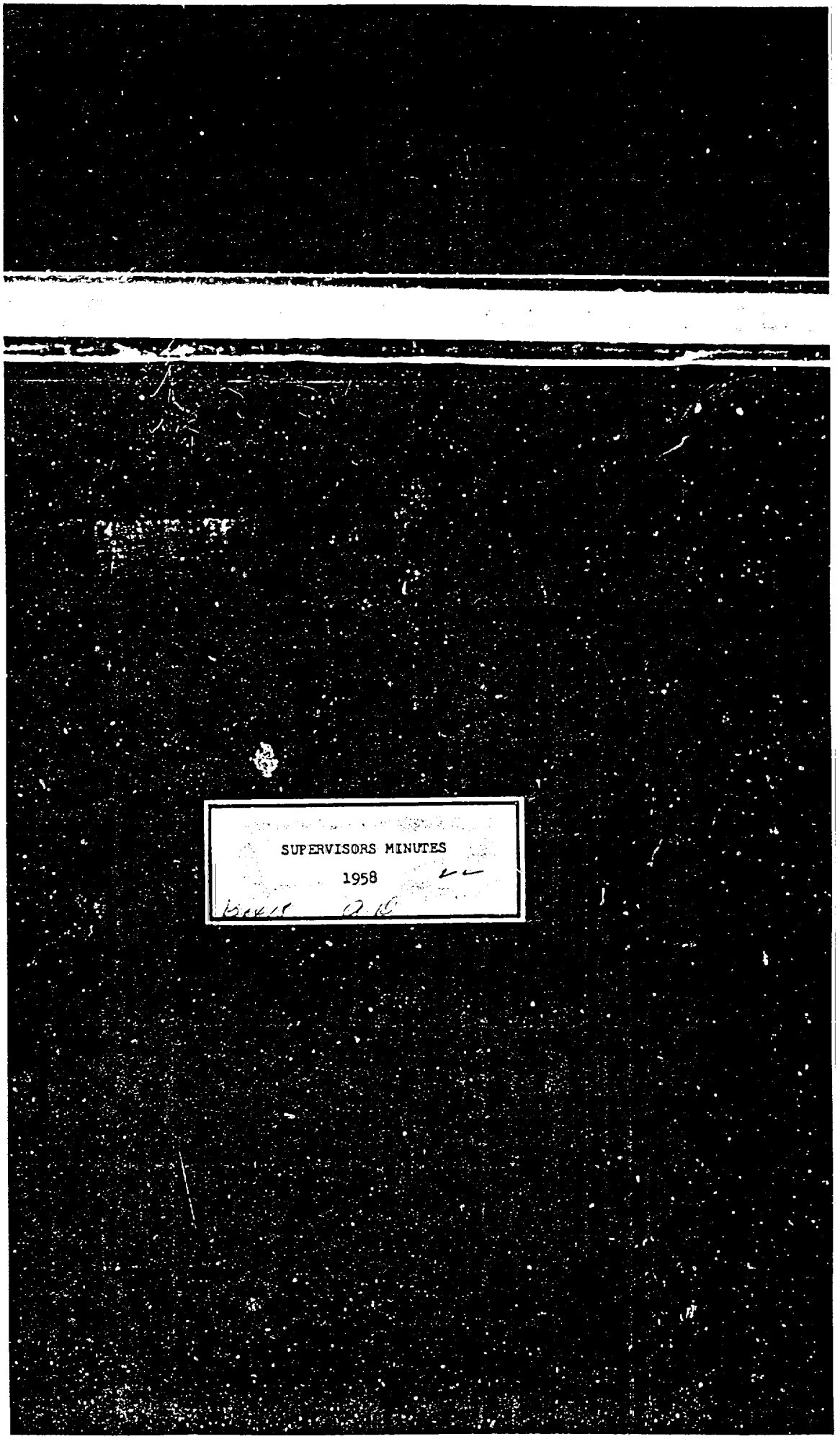

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with cigarettes which have also been checked for resistance to draw to determine this effect upon dynamic burning time.

74-6901 Special Investigations (Butler)

A microscopic examination performed by the development Division showed the first burning Kent paper to be physically identical to Ecusta 515. A chemical investigation is now being made.

Single results on measuring the pH of cigarette smoke gave a range from 5.1 for Viceroy to 5.8 for Parliament. Further tests would be needed to determine if significant differences exist between the results.

Phosphate analyses are complete and running a known sample gave a recovery value of 100.2%.

/hj
cc: Dr. R. N. DuPuis
Dr. S. M. Cantor
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